

PAPER • OPEN ACCESS

Comparative analysis of the structure and internal stress in Ti-6Al-4V alloys manufactured by 3D printing and processing with screw extrusion

To cite this article: N Kazantseva *et al* 2018 *J. Phys.: Conf. Ser.* **1115** 042007

View the [article online](#) for updates and enhancements.



IOP | ebooks™

Bringing you innovative digital publishing with leading voices to create your essential collection of books in STEM research.

Start exploring the collection - download the first chapter of every title for free.

Comparative analysis of the structure and internal stress in Ti-6Al-4V alloys manufactured by 3D printing and processing with screw extrusion

N Kazantseva^{1,2}, I Ezhov¹, N Vinogradova¹, D Davidov¹, A Fefelov³, A Merkushev³, M Ilyinikh³ and A Volkov⁴

¹Institute of Metal Physics UD RAS, 18 Sofia Kovalevskaya Str., Ekaterinburg, 620219, Russia

²Ural Federal University, 19 Mira Str., Ekaterinburg, 620002, Russia

³Regional Engineering Center of Laser and Additive Technology, Ural Federal University, 19 Mira Str., Ekaterinburg, 620002, Russia

⁴Scientific and Production Company Ruthenium Ltd., Ekaterinburg, Russia

E-mail: kazantseva-11@mail.ru

Abstract. Study is based on the compare characterization of the structure and evaluation of the residual internal stresses in Ti-6Al-4V samples manufactured by the 3D printing (selective laser melting) and in the Ti-6Al-4V samples obtained with using of the severe plastic deformation by screw extrusion. The microstructure and residual stresses were examined by transmission electron microscope and X-ray diffractometer. High tensile internal residual stresses in the 3D printed sample were found. The high compressive residual internal stresses were found in the hot pressed and in the twisted samples. It was shown that arising of the high residual stresses in the studied samples under various technological processes occurred in various ways. The residual stresses in the severe plastic deformed samples arose due to non-uniform volumetric plastic deformation. In 3D printed sample, the residual tensile stresses arose from both phase (martensitic) transformation and thermal deformation.

1. Introduction

One of the main tasks of modern developments in the production of titanium alloys is the expansion of the spectrum of metal properties using various processing methods. It is known that metals subjected to strong deformations can acquire completely new properties, most of which can be of practical interest. For example, they may combine high plasticity with great strength. Severe plastic deformation is non-uniform process, which introduces a large number of defects into the structure of the material and promotes the arising of the residual internal stresses. The level and sign of the residual internal stresses (tensile or compressive) affect the mechanical properties of the materials [1]. The method of reverse and comprehensive extrusion with the use of screw extrusion includes several stages of ingot processing and allows one to treat the large-sized ingots [2]. In this case, it is important to know the value of the residual internal stresses after different stages of such treatments to evaluate and predict the mechanical properties of the deformed materials.

Additive technology is a novel surface engineering technique, which allows us to obtained Ti-6Al-4V alloys with high density (about 99.9%) as cast materials [3]. The structure features and laser effect in



3D printing process are currently central and stay under study now. It was found that the 3D printed sample subjected the thermal deformation during building process that provided to the high residual internal stresses in the structure [4, 5].

The main objective of this research is the comparison study the residual internal stresses, causes of their origin, and the ways of structural relaxation in the samples manufactured by the 3D printing (selective laser melting) and in the samples obtained with using of the severe plastic deformation by reverse and comprehensive extrusion.

2. Methodology

The Ti-6Al-4V (ELI) powder produced by TLS Technik GmbH & Co. was taken for the study. The chemical composition of the powder corresponded to ASTM B348 (Grade 23) standard for medical applications. Horizontal Ti-6Al-4V samples were produced by the EOSINT M280 machines (EOS GmbH) equipped with an Ytterbium fibre laser operating at 1075 nm wavelength (IPG Photonics Corp.). The thickness of the powder layer was 30 μm , a back and force scanning scheme was used in an argon atmosphere with hatch distances of 100 μm , the oxygen level in the chamber was 0.07–0.12%. The laser power and scan speed were selected according to the standard installation mode EOSINT M280 for titanium alloys. The industrial balm Ti-6Al-4V samples were used for severe plastic deformation by screw extrusion. After electric high-speed heating, the ingot was twisted by n -turns; the twisted sample was hot pressed with two punches [6]. Schematic picture of the twisting and pressing the twisted sample into a round washer is presented in figure 1 [2].

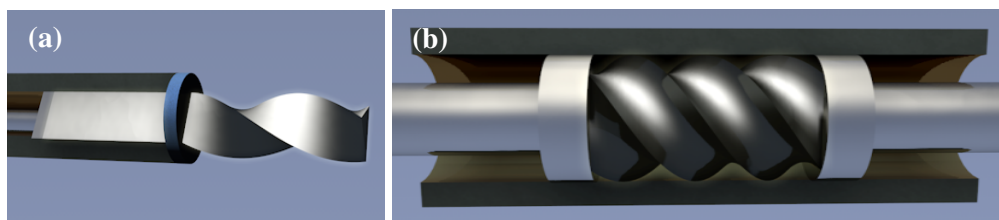


Figure 1. Severe plastic deformation of the samples: (a) screw extrusion of the samples; (b) hot pressing a twisted blank into a washer.

As a reference, we used a sample, which was cut from a rod of an industrial medical alloy Ti-6Al-4V (ELI) (ASTM F136, Grade 5). The study was carried out using an optical microscope Micromed MET, a scanning electron microscope JSM 6490 with the energy dispersive and wave micro-analyser Oxford Inca, transmission electron microscopes JEM-200CX, Tecnai G2 30 Twin, and X-ray diffractometer DRON-3 with $\text{CuK}\alpha$ radiation. The mechanical properties were measured at room temperature using a Nano Test nanoindenter, with a maximum test load of 32 mN and a load application time of 10 sec.

3. Results and discussion

X-ray diffraction patterns of the reference sample and samples after severe plastic deformation (twisted and pressed) show two phase state with the alpha (HCP) and beta (BCC) phases. The reference sample (Ti-6Al-4V ELI) exhibits an α grain size of 1 μm and a β grain size of approximately 0.45 μm with a proportion of 94% of α phase and 6% of β phase. The grain size in the sample after pressing is decreased as it is compared to the sample after twisting. On the X-ray diffraction patterns of the 3D printed sample only the lines of the HCP phase are present. No diffraction reflections of metastable phases or cubic β -phase were detected. The 3D printed sample shows a structure with a grain size of 200 μm (table 1).

The X-ray diffraction lines of the studied samples are shifted relative to the positions of the reference lines. This fact indicates the presence of residual stresses (thermal) in the volume of the material. High tensile residual stresses are found in the 3D printed sample. The high compressive residual stresses are found in the deformed samples (table 2). As can be seen from the table 2, the values of the compressive

residual internal stresses in the deformed samples are close to each other. One can say that 3D laser printing process produces other thermal stresses as it is compared with that in hot screw extrusion or hot pressing.

Table 1. Crystal parameters of HCP phase and average grain size in the studied samples.

Samples	a , nm ± 0.0002	c , nm ± 0.0002	c/a	Phase composition	Average grain size μm
Reference	0.2922	0.4673	1.599	HCP+BCC	1
3D printed	0.2928	0.4672	1.596	HCP	200
Twisted	0.2924	0.4681	1.600	HCP+BCC	1900
Pressed	0.2924	0.4679	1.600	HCP+BCC	730

Similar high tensile residual stresses were also found in a 3D printed sample with horizontal building orientation in [4]. High tensile residual stresses are undesirable in the material and can lead to a spontaneous change in the dimensions of the product during its operation or storage due to the gradual redistribution of residual stresses during their relaxation [1]. Uncontrolled tensile residual stresses in the volume of the material can be compensated for by controlled surface compressive stresses. This process is usually performed by means of various surface treatments, for example, ultrasonic, laser treating [7]. Residual surface stresses were calculated from the following formula [8]: $H_{IT} - H_{IT,0}$, where $H_{IT,0}$ is the hardness of the reference sample, obtained with nanoidentor. In our case, the plastic deformation by hot pressing provides the compressive residual stresses in volume of the material and on the surface (table 2). As can be seen from the table 2, the 3D printed sample shows the highest elastic modulus and nanohardness in comparison with the reference and deformed samples. This fact rather concerns with the structure morphology and phase composition of the studied samples than their grain sizes. The pressed sample shows the lowest elastic modulus. Nanohardness of this sample is close to one for twisted sample, in spite of that this sample has the less grain size than the twisted sample.

Table 2. Elastic modulus and residual internal stresses of the studied samples.

Samples	Elastic modulus, GPa	HV, GPa	Residual internal stresses, MPa	Residual surface stresses, MPa
Reference	132	5.3	-	-
3D printed	149	6.3	458	1000
Twisted	140	4.6	-190	-700
Pressed	127	4.3	-215	-1000

High level of internal residual stresses in the structure of the studied samples can be observed by TEM. Figure 2 shows two phase structure of the reference sample. This structure has regions with plates of the HCP α -phase and small regions of the BCC β -phase between the HCP regions. Transmission electron microscopic (TEM) studies of 3D samples shows the twinned martensitic α' - phase structure without any β precipitations (figure 3). These twins are the (10-12) HCP tensile twins. Similar twins were found in [3] in the Ti-6Al-4V (ELI) alloy manufactured by the 3D printing (selective laser melting). Such type of the twins indicates the presence of tensile stresses in the sample. In differ from the 3D printed sample, the twisted sample does not show any twins. The structure of the twisted sample has the elongated areas with the plates of the HCP α -phase inside of them and the thin oblong BCC β -phase layers between the HCP regions (figure 4b). We observed the fragmentation of the α -plates in this sample. Elongated BCC β -phase layers between HCP α -phase regions are also observed in the structure of the pressed sample (figure 4cd). The HCP α plates form in accordance with the orientation relationship between BCC and HCP crystal lattices [9]:

$$\begin{pmatrix} u \\ v \\ w \end{pmatrix}_{HCP} = \begin{pmatrix} \bar{1} & 1 & 1 \\ 1 & \bar{1} & 1 \\ 1 & 1 & 0 \end{pmatrix} \cdot \begin{pmatrix} u \\ v \\ w \end{pmatrix}_{BCC}.$$

High defect density in the structure may be noted with the presence the diffuse scattering in the SAED patterns of this sample.

As we cut the deformed samples from the center of the pressed rod, the founded internal compressive stresses were not the surface stresses. It means that the hot pressing promotes the relaxation of the residual tensile stresses in the volume of the material and accumulation of new defects in the structure of the sample.

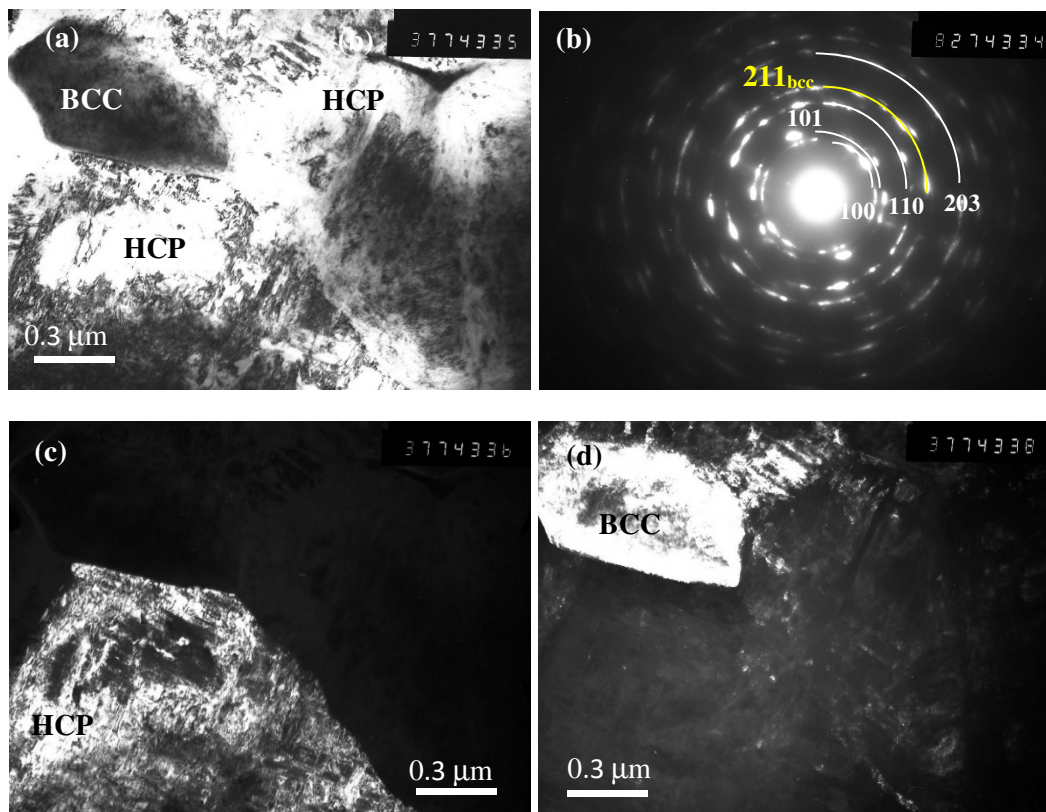


Figure 2. Microstructure of the reference, TEM: (a) the bright-field image; (b) SAED pattern to (a)-(d); (c) the dark-field image in HCP phase reflex; (d) the dark-field image in BCC phase reflex.

Residual stresses in the materials arise under the influence of non-uniform cooling or heating, cold work or phase and structural transformations. Their occurrence are based on irreversible volumetric changes in the material. Under deformation (hot, warm or cold), the main reasons for occurrence of the residual stresses are inhomogeneous distributions of deformations and temperatures in the volume of the sample [1].

In our case, we can say that the formation of residual internal stresses under various technological processes occurs in the different ways. In 3D printed sample, the residual tensile stresses arise under both phase transformation (martensitic transformation) and thermal deformation. Fast cooling of the printed layers by ambient powder is cause of the martensitic transformation. Temperature gradient in the sample manufactured by the selective laser melting was found in [9]. As it was shown by authors of [9], the temperature in the different parts of the 3D printed sample cyclically could be changed from 600 and to 2900 K. Extension under heating and compression under cooling of the part the sample leads to its thermal deformation. In our sample, the presence of non-uniform distribution of the deformation may

be tracked by the local twinning. Because, the twinning does not depend on the temperature and takes a special level of the stresses.

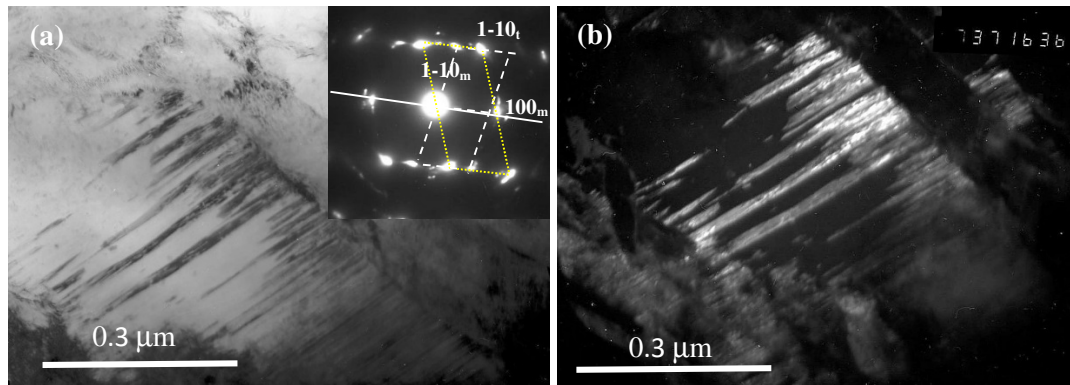


Figure 3. Microstructure of the 3D printed sample, TEM: (a) the bright-field image; (b) the dark-field image in HCP twin reflex, zone axis $[001]_m$, (10-12) twin plane.

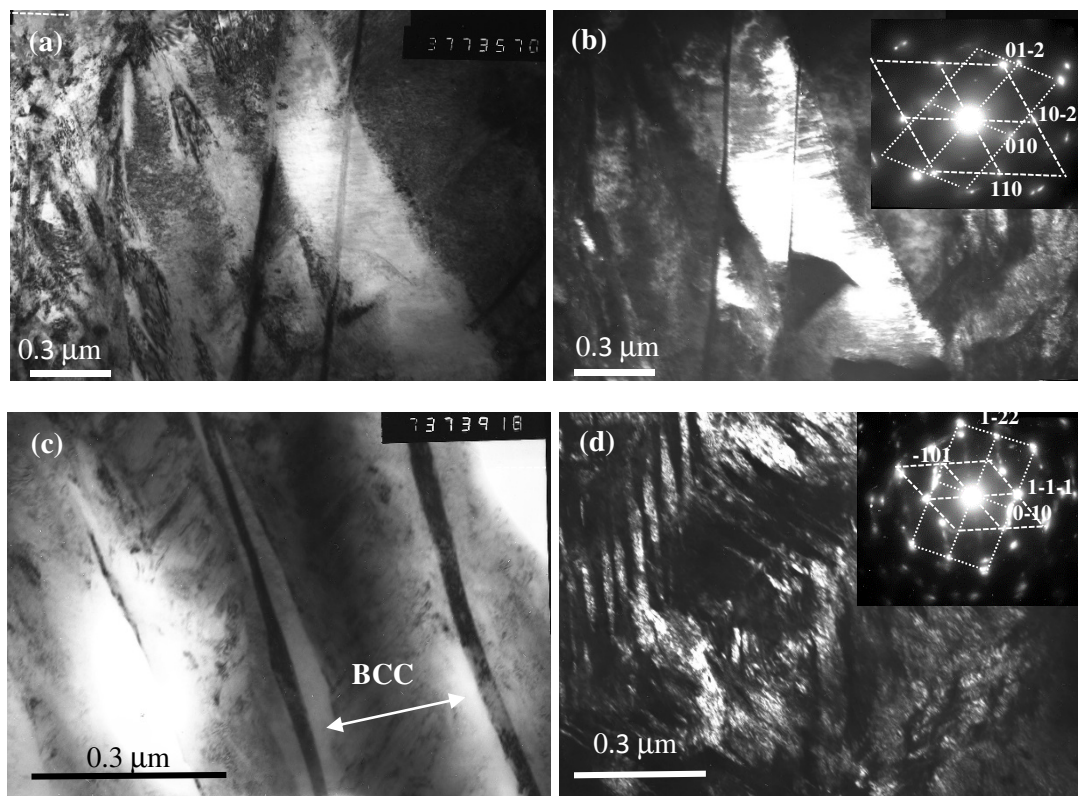


Figure 4. Microstructure of the deformed samples, TEM: (a)–(b) twisted sample, (a) the bright-field image, zone axis: $[021]$, $[2-21]$, (b) the dark-field image in (01-2)HCP phase reflex; (c)–(d) pressed sample, (c) the bright-field image, zone axis $[101]_{HCP}$, $[121]_{HCP}$, (d) the dark-field image in $(-111)_{HCP}$ reflex.

The residual compressive stresses in the severe plastic deformed samples arise under non-uniform volumetric plastic deformation. Absence of the twinning in the twisted or compressed samples may be associated with deformation texture, which is not favour for twinning. Effect of the texture on the twinning in the Ti-6Al-4V alloy was studied in [11, 12], where the Schmidt factors for all twinning

systems (compressive and tensile) were calculated. Tensile {10-12} twinning was correlated with the maximum Schmidt factor for <1-100>HCP loading direction [12].

4. Conclusion

The following conclusions can be done from this study:

1. The microstructure and residual stresses in the 3D printed sample and samples after severe plastic deformation by reverse and comprehensive extrusion were studied.
2. It is found the high tensile residual stresses in the 3D printed sample. The high compressive residual stresses are found in the hot and in the twisted pressed samples.
3. It is shown that the formation of the high residual stresses in the studied samples under various technological processes occurs in various ways. The residual stresses in the severe plastic deformed samples arise due to non-uniform volumetric plastic deformation. In 3D printed sample, the residual tensile stresses arise from both the phase (martensitic) transformation and thermal deformation.

Acknowledgments

This work was supported by Russian Found of Basic Research N 17-03-00084, State programs: "Diagnostics" AAA-A18-118020690196-3 and "Pressure" AAA-A18-118020190104-3. TEM studies were done in the Centre of collective usage of the Institute of Metal Physics, Ural Branch of Russian Academy of Sciences.

References

- [1] Burkin S P, Shimov G V, Andrukova E A 2015 *Residual stresses in metals* (Ekaterinburg: Ural Federal University) p 12
- [2] Volkov A E 2015 *Journal Titan* **1** 35
- [3] Krakhmalev P, Fredricsson G, Yadroitseva I, Kazantseva N, Du Plessis A and Yadroitsev I 2016 *Physics Procedia* **83** 778
- [4] Kruth J-P, Froyen L, Van Vaerenbergh J, Mercelis P, Rombouts M and Lauwers B 2004 *Journal of Materials Processing Technology* **149** 616
- [5] Zyl I, Yadroitsava I and Yadroitsev I 2016 *South African Journal of Industrial Engineering* **27** 134
- [6] Volkov A E 2013 RF Patent N 2013129568
- [7] Novikov I I 1978 *Theory of heat treatment* (Moscow: Metallurgy) p 89
- [8] GOST R 57172-2016 *Definition of surface residual stresses by the indentation method* (M: Standardinform, 2016 in Russian)
- [9] Seward G G E, Celotto S, Prior D J, Wheeler J and Pond R C 2004 *Acta Materialia* **52** 821
- [10] Yadroitsev I and Yadroitsava I 2015 *Virtual and Physical Prototyping* **10** 67
- [11] Kazantseva N, Krakhmalev P, Yadroitsev I, Fefelov A, Vinogradova N, Ezhov I and Kurennykh T 2017 *International Journal of Materials and Metallurgical Engineering* **11** 762
- [12] Kazantseva N V, Krakhmalev P V, Fredriksson G, Vinogradova N I, Yadroitsev I A and Ezhov I V 2017 *Journal Titan* **56** 8